AD-750 593

HOT HARDNESS INVESTIGATION OF REFRACTORY METALS AND ALLOYS

Cyrus H. Philleo, et al

Army Weapons Command
Rock Island, Illinois

September 1972

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SWERR-TR-72-63

HOT HARDNESS INVESTIGATION OF REFRACTORY METALS AND ALLOYS

TECHNICAL REPORT

Cyrus H. Phillea

and

Donald H. Sale

September 1972

RESEARCH DIRECTORATE
WEAPONS LABORATORY, WECOM
RESEARCH, DEVELOPMENT AND ENGINEERING DIRECTORATE
U. S. ARMY WEAPONS COMMAND

30

Approved for public release, distribution unlimited

R

Unclassified

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)

U. S. Army Weapons Command
Research, Dev. and Eng. Directorate
Rock Island, Illinois 61201

2a. REPORT SECURITY CLASSIFICATION: Unclassified

2b. GROUP

3. REPORT TITLE

HOT HARDNESS INVESTIGATION OF REFRACTORY METALS AND ALLOYS (U)

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

5. AUTHOR(S) (First name, middle initial, last name)

Cyrus H. Philleo and Donald H. Sale

6. REPORT DATE: September 1972

7a. TOTAL NO. OF PAGES: 34

7b. NO. OF REFS: 7

8a. CONTRACT OR GRANT NO.

b. PROJECT NO. DA 1T062105A328

c. AMS Code 502E.11.294

d.

9a. ORIGINATOR'S REPORT NUMBER(S): SWERR-TR-72-63

9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report)

10. DISTRIBUTION STATEMENT

Approved for public release, distribution unlimited.

11. SUPPLEMENTARY NOTES

Details of illustrations in this document may be better studied on microfiche.

12. SPONSORING MILITARY ACTIVITY

U. S. Army Weapons Command

13. ABSTRACT

This investigation was conducted by the Research Directorate, Weapons Laboratory, WECOM, to determine the hardness of several refractory metals and alloys at temperatures from 70°F to 2000°F. Microhardness tests were made in vacuum under controlled conditions to provide comparable screening data for predicting the strengths of these materials in high-temperature applications such as gun barrel liners. Materials tested included tungsten, molybdenum, columbium, tantalum, columbium-base alloys, and tantalum-base alloys. Chromium plate and Cr-Mo-V steel, currently used in gun barrels, were also tested for comparison. Several of the refractory alloys retained most of their room-temperature hardness up to 1800°F or 2000°F. Some were harder at these temperatures than at 1000°F. Chromium plate was harder than any of the refractory metals and alloys up to about 1100°F, but was softer than most at 1500°F and above. Cr-Mo-V steel was softer at 1800°F than any of the other materials tested. The three alloys showing the highest hardness values at 1800°F and above were: (a) Su-16, a columbium-base alloy; (b) Ta-111, a tantalum-base alloy; and (c) Ta-222, a tantalum-base alloy. (U) (Philleo, C. H. and Sale, D. H.)

DD FORM 1 NOV 65 1473 REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

Unclassified)

Security Classification

Unclassified

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
1. Hot hardness test						
2. Refractory metals						

Unclassified

Security Classification

AD

RESEARCH DIRECTORATE

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ILLUSTRATIONS

OBJECTIVE

The objective of this investigation was to provide comparative hot hardness data for a number of refractory metals and alloys to assist in the selection of materials for use in gun barrels for automatic weapons.

BACKGROUND

The trend in newer weapon designs is toward higher cyclic firing rates, lighter weight, and high performance under sustained firing. These requirements produce conditions that exceed the high temperature resistant capabilities of the conventional steels used for weapon construction. To obtain heat resistance and strength at higher temperatures requires consideration of refractory metals such as molybdenum, columbium, tantalum and tungsten, and their alloys.

Data on the refractory metals are incomplete with regard to properties such as hardness at high temperatures. This work was undertaken to provide preliminary data that would have a common comparison basis and would provide an initial ground for selection of metals that appear promising for further exploration as weapon materials for such applications as machine gun barrels.

APPROACH

All refractory metals and alloys selected and obtained were all commercially available materials. For comparison, specimens were also obtained of chromium plate (over chromium-molybdenum-vanadium steel) and of a chromium-molybdenum-vanadium steel gun barrel section. The materials compared are listed below:

1. Chromium. (as plated on gun barrel steel)

2. Tungsten.

3. Tantalum. 9% tungsten, 2.50% hafnium, 0.01% carbon (Ta-222)

4. Tantalum. 9.6% tungsten, 2.4% hafnium, 0.01% carbon (Ta-111)

5. Molybdenum.

6. Columbium. 11% tungsten, 3% molybdenum, 2% hafnium, 0.08 carbon (Su-16)

7. Columbium. 33% tantalum, 1% zirconium, annealed to Rockwell A39 hardness.

8. Columbium. 33% tantalum, 1% zirconium, annealed to Rockwell A46 hardness.

9. Columbium. 10% tungsten, 10% tantalum.

10. Tantalum.

11. Columbium.

12. Chromium-Molybdenum-Vanadium steel (per cent)

Carbon,	0.41-0.49
Manganese,	0.60-0.90
Phosphorous,	0.40 max
Sulfur,	0.040 max.
Silicon,	0.20-0.35
Chromium,	0.80-1.15
Molybdenum,	0.30-0.40
Vanadium,	0.20-0.30
Iron,	Balance

Hardness indentations were made in vacuum (approximately 3×10^{-5} Torr) at a series of successively higher temperatures of 70°F, 500°F, 1000°F, 1500°F, 1800°F, and for some materials, 2000°F. After the test temperature had been reached, the specimen was held at temperature for a minimum of 10 minutes to stabilize before indentations were made. The specimens were cooled to near room temperature in vacuum, then removed from the vacuum chamber for measurement of the indentations in air.

The hot hardness test was used for this study because it has certain advantages over most other mechanical property tests:

1. Specimens are small, simple, and easily prepared.

2. Many tests can be made on a single specimen.

3. Problems of obtaining uniaxial loading and uniform temperature over a specimen are largely eliminated.

Although hardness has no fundamental reference standard (such as pounds per square inch, as used for a tensile test), hot-hardness properties are of considerable value for providing screening data to predict the high-temperature behavior of materials

The hot-hardness test apparatus is shown in Figure 1 The coldwall vacuum chamber has an internal heater shown in Figure 2 A sapphire indenter, shaped to a standard 136° pyramid (Vickers or DPH) was applied to the specimen under a 500-gram deadweight load inside the chamber The load indenter assembly is shown in Figure 3. Load was applied to the specimen for 15 seconds measured with an electrical timer. A pair of electrical contacts was mounted on the balance arm and a bracket on the load assembly in such a manner that they opened when the specimen picked up the full load on the indenter This extinguished a small light bulb connected through the contacts to a 6-volt battery Timing was started at that instant

Application of the load to the specimen was accomplished by means of a motor and worm-gear drive by which the complete indenter system was lowered and raised The speed of the drive was the same for each test, and ensured a uniform, slow, smooth application of the load

The specimen was moved horizontally for successive tests at different temperatures by means of the fixture shown in Figure 2 The rod was connected to the specimen holder extended through O-ring seals in the side of the vacuum chamber and was moved by turning a wingnut on the threaded end of the rod

The indenter was also moved horizontally through an arc by moving the pointer on top of the vacuum chamber which rotated the shaft supporting the load-indenter assembly This permitted making several indentations without moving the specimen

For most of the tests, five or more indentations were made at each test temperature On some materials, however, the combination of vacuum and time at temperature caused etching which made measurement of the indentations difficult or impossible When this occurred, another specimen of the same material was prepared and indentations were measured after testing at each temperature Subsequent tests were always made at higher test temperatures

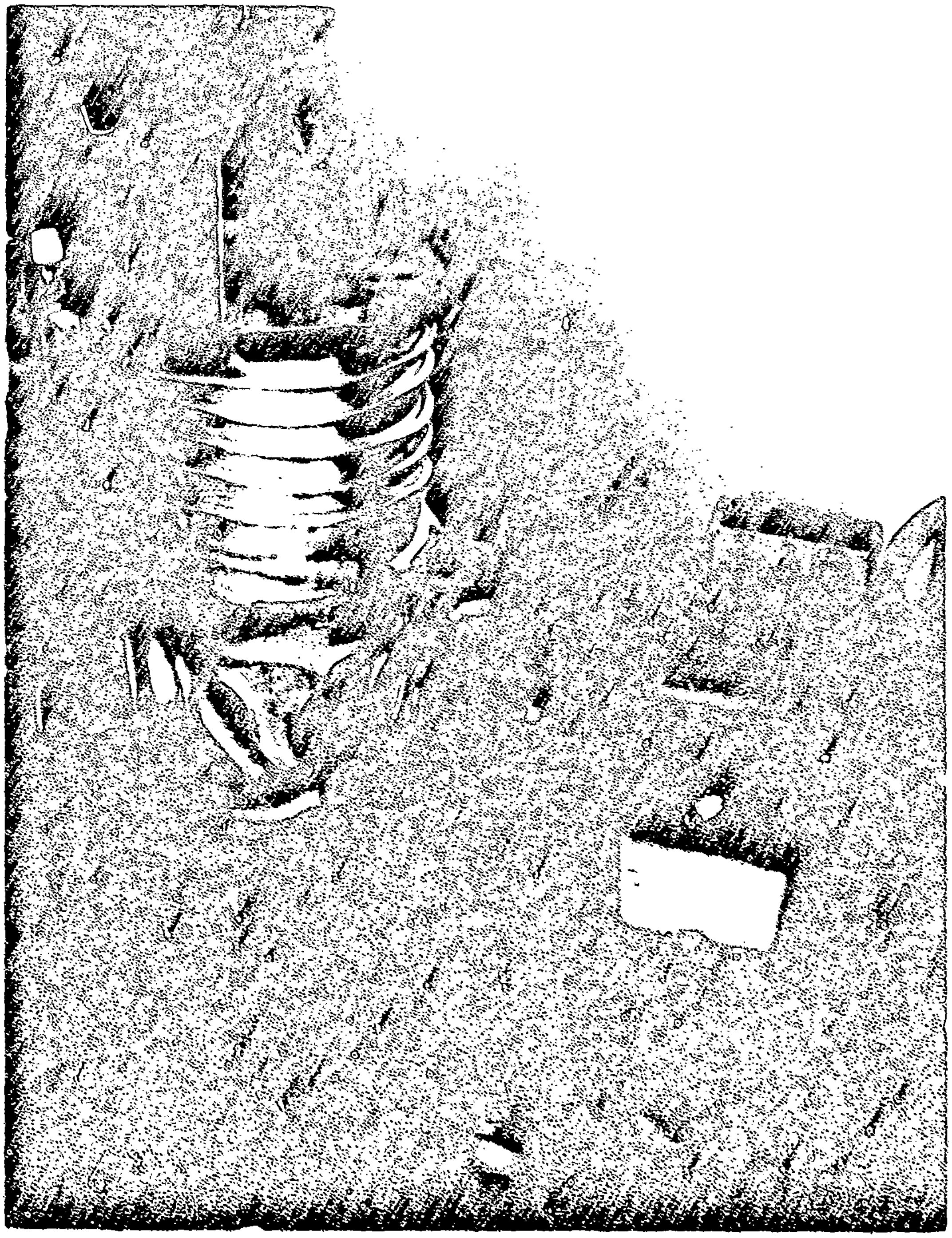


FIGURE 1 Vacuum hot hardness tester

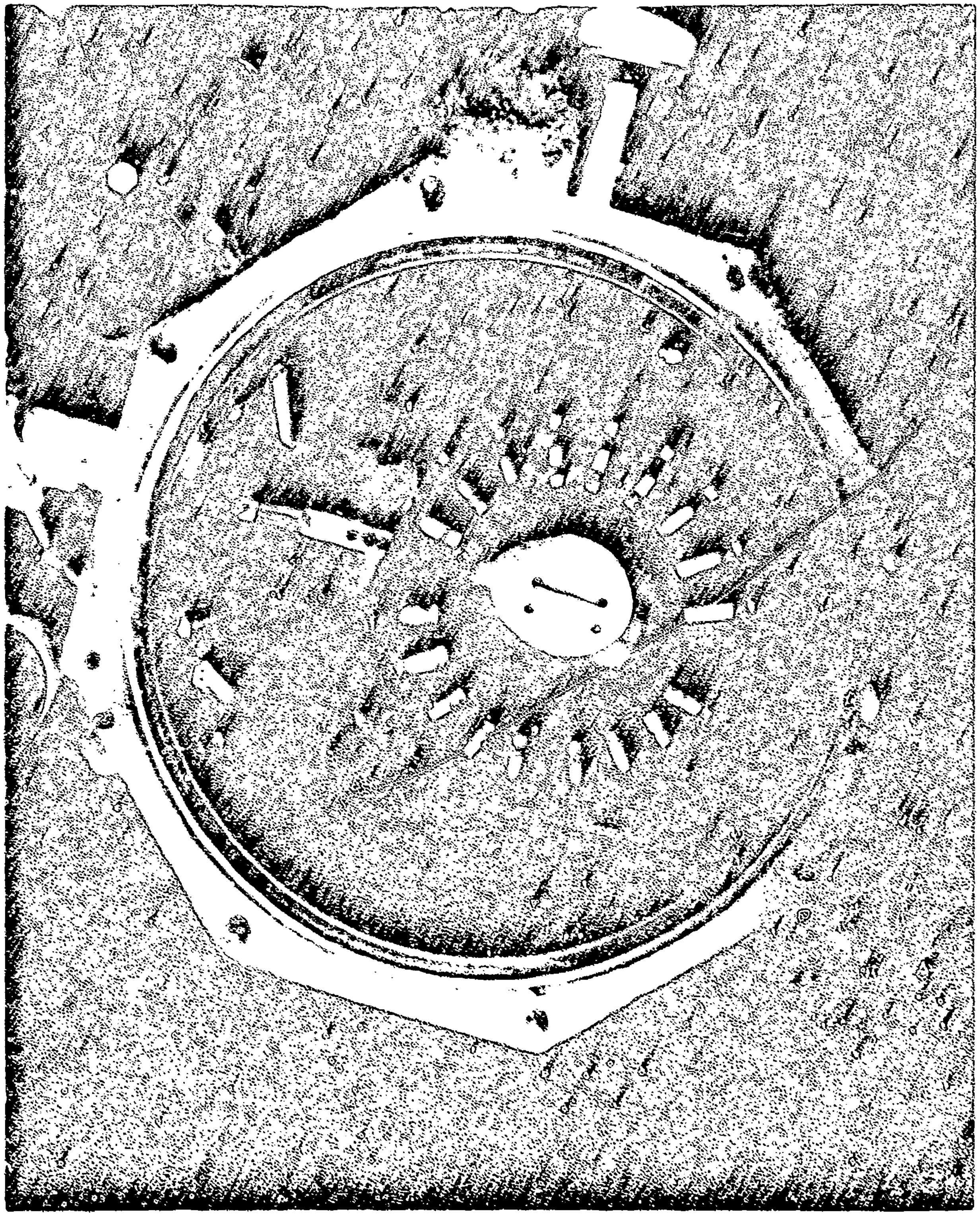


FIGURE 2 View of lower chamber of hot hardness tester showing heater, specimen, and specimen positioning device

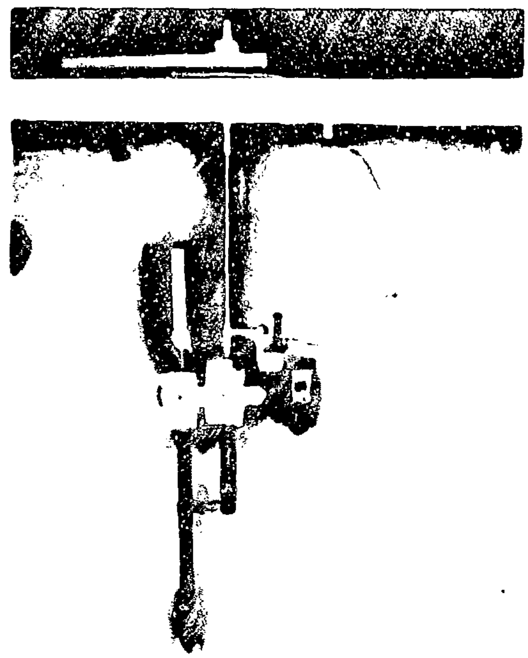


FIGURE 3 Internal load-indenter assembly for hot hardness tester

Measurement of all indentations was done at room temperature in air. Most measurements were made on a Vickers microhardness tester. However, some were made on a metallograph to provide special lighting for indentations otherwise difficult to measure.

The test specimens were 1 inch by 3/8 inch by approximately 1/16 inch. For preparing the specimen surface for hardness tests, each specimen was mounted in Bakelite and polished on a metallographic polishing wheel with diamond polishing compound. The mount was then broken away from the specimen.

A combination of automatic and manual control was used to provide temperature control. A potentiometer-type automatic temperature controller-recorder was set to bring the specimen up to within a few degrees of the desired temperature. A variable transformer in the control circuit was then adjusted to provide the required amount of power to bring the specimen to the desired temperature and hold it there with a minimum of cycling. The thermocouple junction was in the anvil supporting the specimen.

RESULTS AND DISCUSSION

The test results show the effects of temperature on hardness in a nonaggressive environment on the metals tested.

The data show that several of the refractory alloys maintain a comparatively high level of hardness up to 1800°F or above. On the assumption that the hardness-strength relationship at ambient temperature is realistic when temperature rises, these alloys appear promising for use in gun barrel liners provided other requirements are met.

The three alloys which showed the highest hot hardness values at 1800°F are given below:

Su-16. Columbium-base alloy with 11% tungsten, 3% molybdenum, 2% hafnium, and 0.08% carbon; average hardness at 1800°F, 320 DPH (approximately Rockwell C 32).

Ta-111, or tantalum-W-Hf-C. Tantalum-base alloy with 9.6% tungsten, 2.4% hafnium, 0.01% carbon; average hardness at 1800°F, 280 DPH (approximately Rockwell C 27).

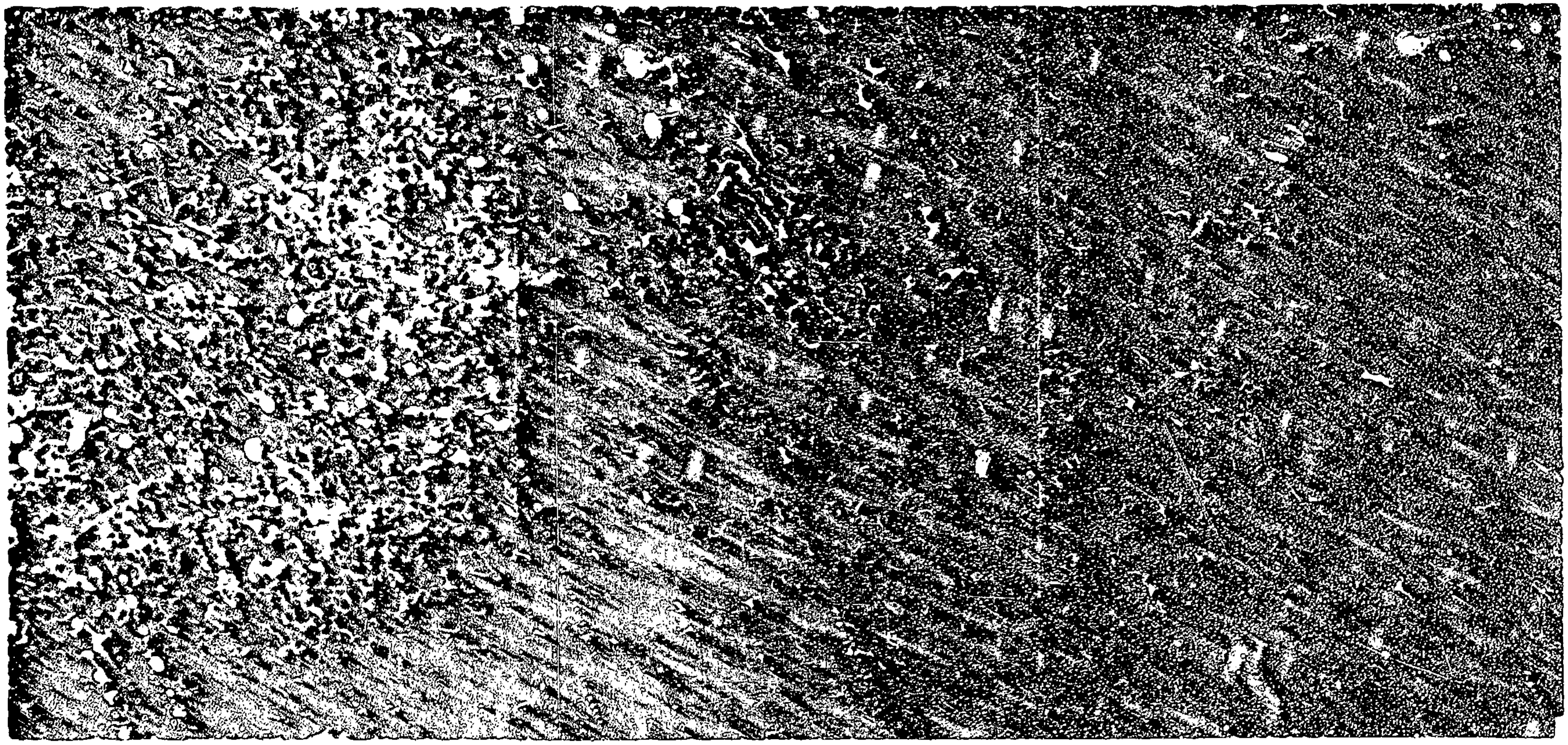
Ta-222. Tantalum-base alloy with 9% tungsten, 2.5% hafnium, <0.0175% carbon; average hardness at 1800°F, 240 DPH (approximately Rockwell C 21).

Some examples of the indentations produced are shown in Figures 4 through 11. Figures 4, 5, and 6 are typical indentations showing how the size increased with increasing temperature. Figure 7 shows the indentation made in tantalum at 2200°F. The tantalum was only slightly etched or discolored after testing to this temperature. The center of the indentation is slightly out of focus because of the shallow depth of focus on the metallograph at this magnification.

Because of vacuum etching and heat effects, some indentations were difficult or impossible to measure under standard bright-field illumination. In many instances, the indentation could be measured with dark-field illumination on the metallograph. Figures 8, 10, and 11 are photomicrographs taken with dark-field illumination. Figure 9 was made with bright-field illumination, but with the aperature diaphragm off center to provide side lighting.

The hardness-versus-temperature plots of all the materials tested are given together in Figure 12 and individually in Figures 13 through 24. Because chromium was much harder at room temperature (DPH 940, Rockwell C 68) than any of the other materials tested, the hardness coordinate for chromium (Figure 13) and for the composite plot (Figure 12) is not the same as that used for the other materials (Figures 14 through 24).

Although the hardness of the electroplated chromium dropped quite rapidly with increasing temperature, it was harder than any of the other materials tested up to about 1100°F (Figure 12) The hardness at 1100°F was 225 DPH, (approximately Rockwell C 18). Gun barrel bores are presently chromium-plated to reduce wear and extend barrel life. Figure 9 shows a chromium deposit after testing at 1000°F. It appeared about the same as it did at room temperature. The crack pattern is normal and existed before any tests were made at elevated temperatures. Figure 10 shows the same specimen after testing at 1500°F. Dark-field illumination was used here to make the indentations more visible. Three indentations can be seen in this photograph. Figure 11 shows chromium plate after testing at 1800°F (also with dark-field illumination). The specimen showed considerable spalling of the chromium and had many iridescent areas. Spalling of the chromium plate was of interest in that it occurred without applied stresses other than thermal effects.



Room Temperature | 1000F | 2000F

Figure 4 | Figure 5 | Figure 6

FIGURES 4, 5 & 6 Photomicrographs of electron-beam-melted columbium, showing DPH indentations made at room temperature, 1000F, and 2000F. 50X

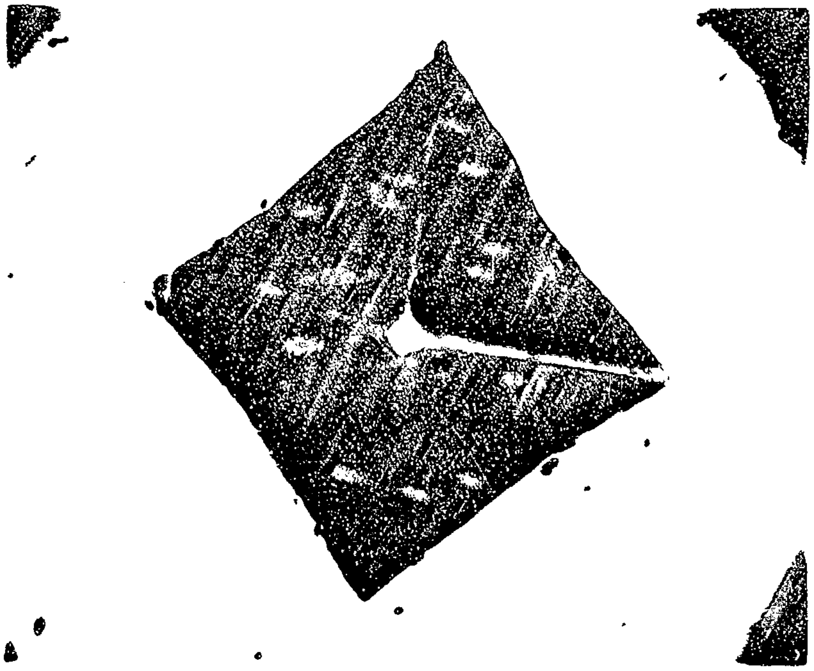


FIGURE 7 DPH indentation made at 2200F in unalloyed tantalum. 300X

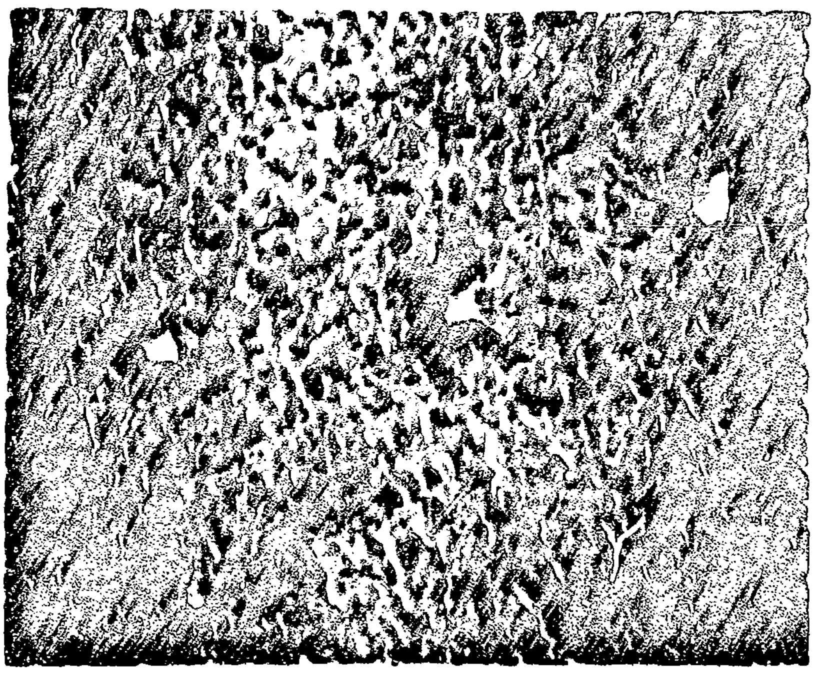
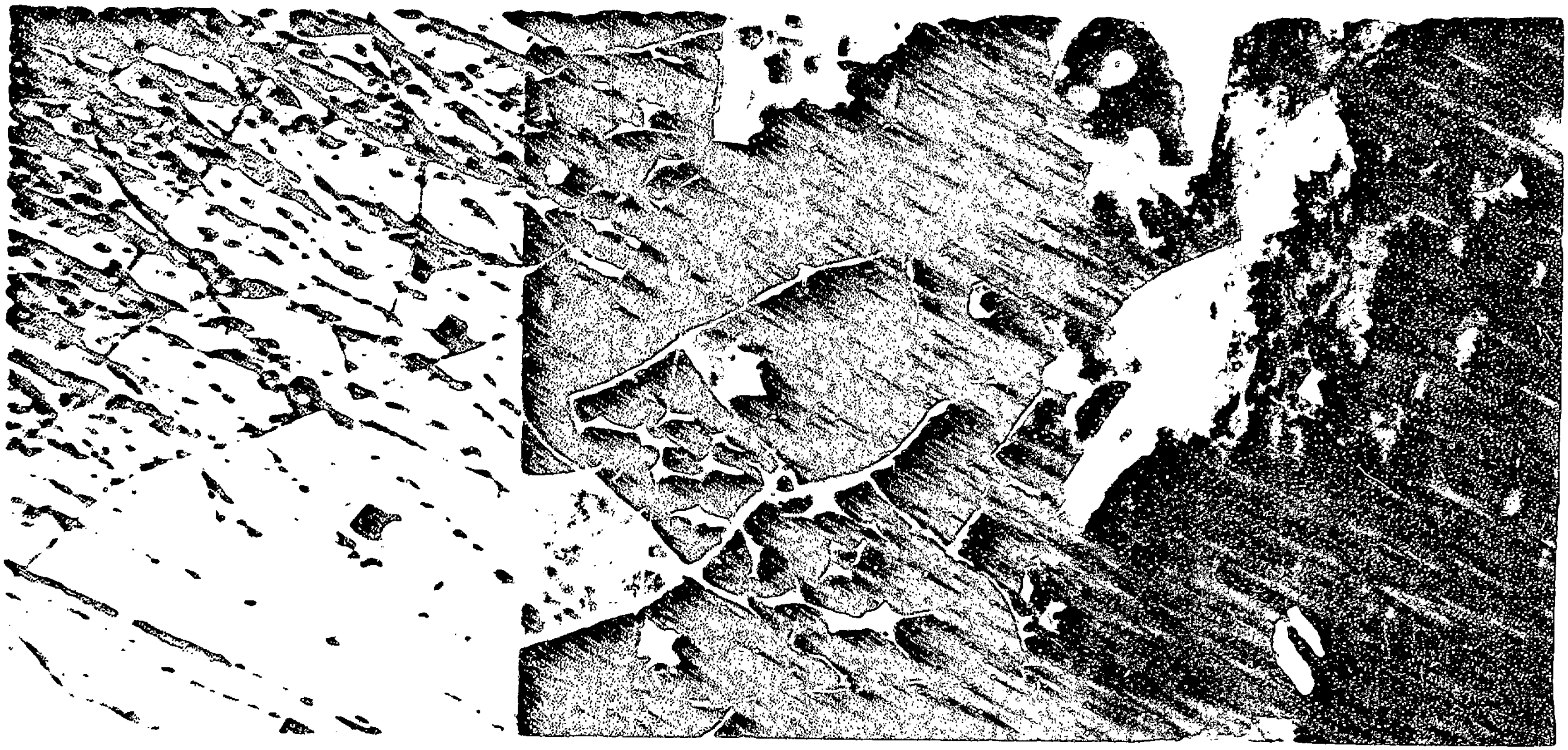


FIGURE 8 Electron-beam-melted columbium showing DPH indentations made at 1800F. Note vacuum etching. 100X. Dark field illumination.

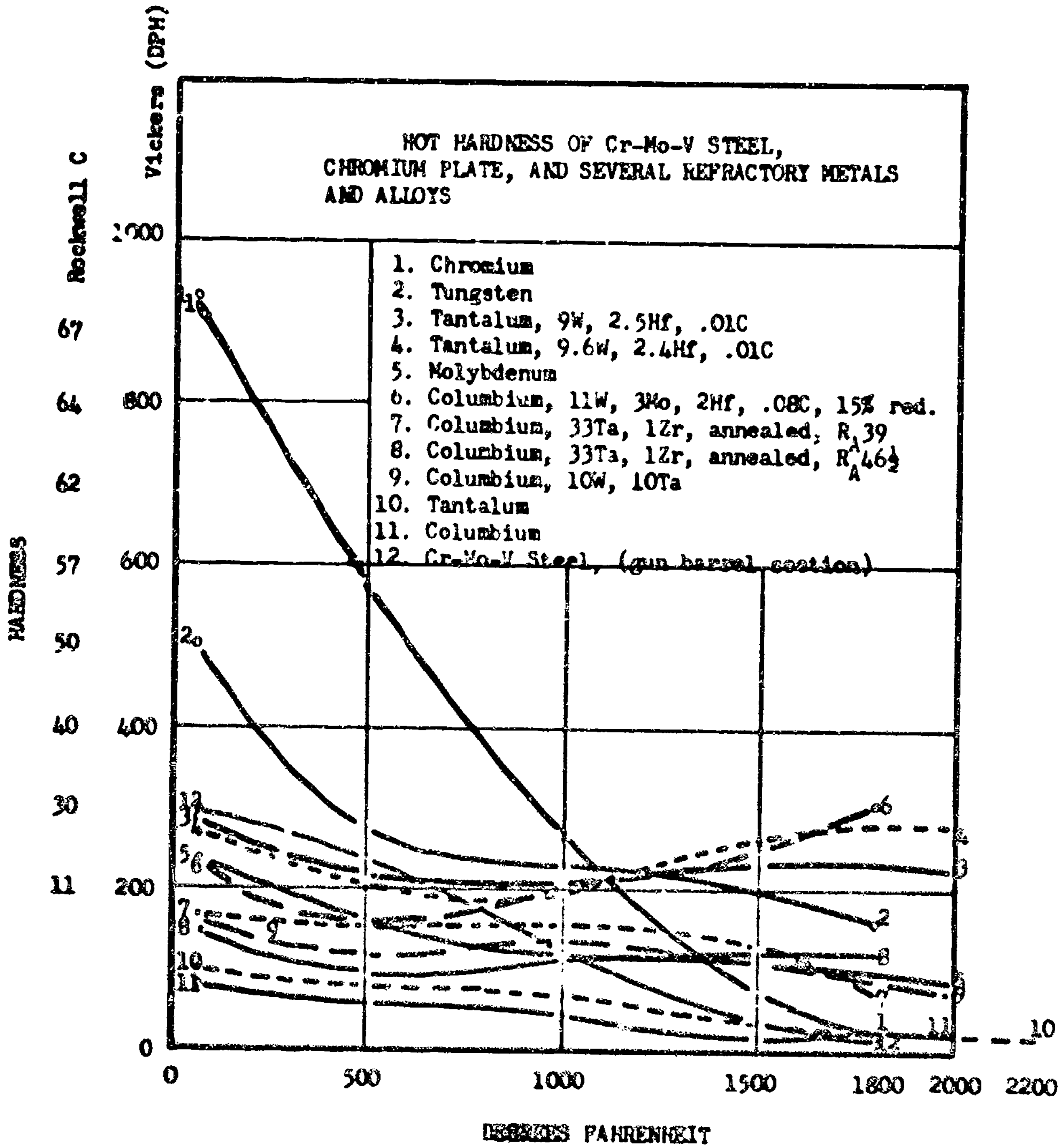


1000F
Figure 9

1500F
Figure 10

1800F
Figure 11

FIGURES 9, 10, & 11 Chromium plate after testing at 1000F, 1500F, and 1800F. Figure 9 was made with bright field illumination and aperature off center for side lighting. Figures 10 & 11 had dark field illumination. All are 100X magnification.



HOT HARDNESS OF Cr-Mo-V STEEL, CHROMIUM PLATE, AND SEVERAL REFRACTORY METALS AND ALLOYS
1. Chromium
2. Tungsten
3. Tantalum, 9W, 2.5Hf, .01C
4. Tantalum, 9.6W, 2.4Hf, .01C
5. Molybdenum
6. Columbium, 11W, 3Mo, 2Hf, .08C, 15% red.
7. Columbium, 33Ta, 1Zr, annealed, R_A39
8. Columbium, 33Ta, 1Zr, annealed, R_A46½
9. Columbium, 10W, 10Ta
10. Tantalum
11. Columbium
12. Cr-Mo-V Steel, (gun barrel section)
HARDNESS
Rockwell C
Vickers (DPH)
DEGREES FAHRENHEIT

FIGURE 12

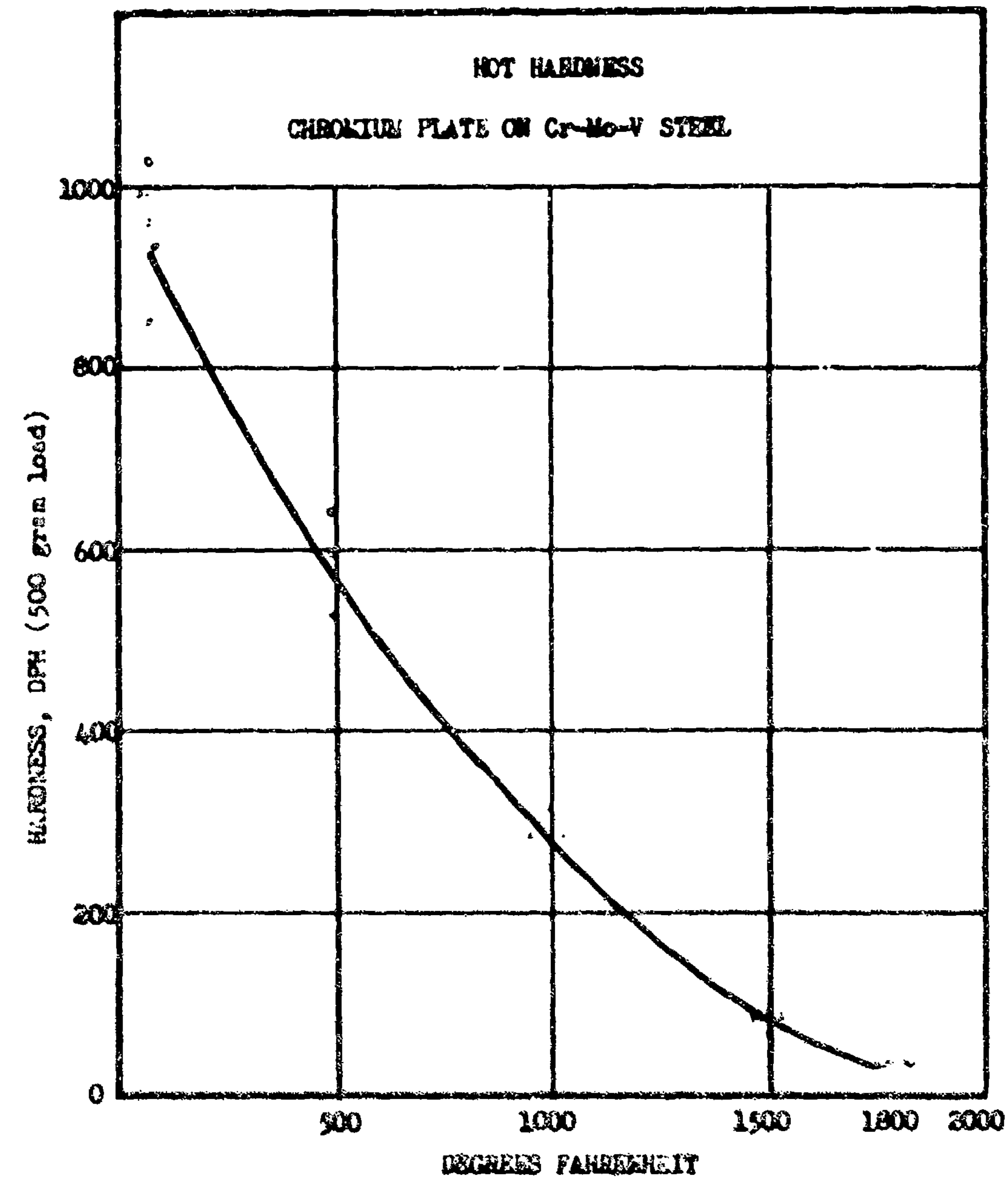

HOT HARDNESS
CHROMIUM PLATE ON Cr-Mo-V STEEL
HARDNESS, DPH (500 gram load)
1000
800
600
400
200
0
500
1000
1500
1800
2000
DEGREES FAHRENHEIT

FIGURE 13

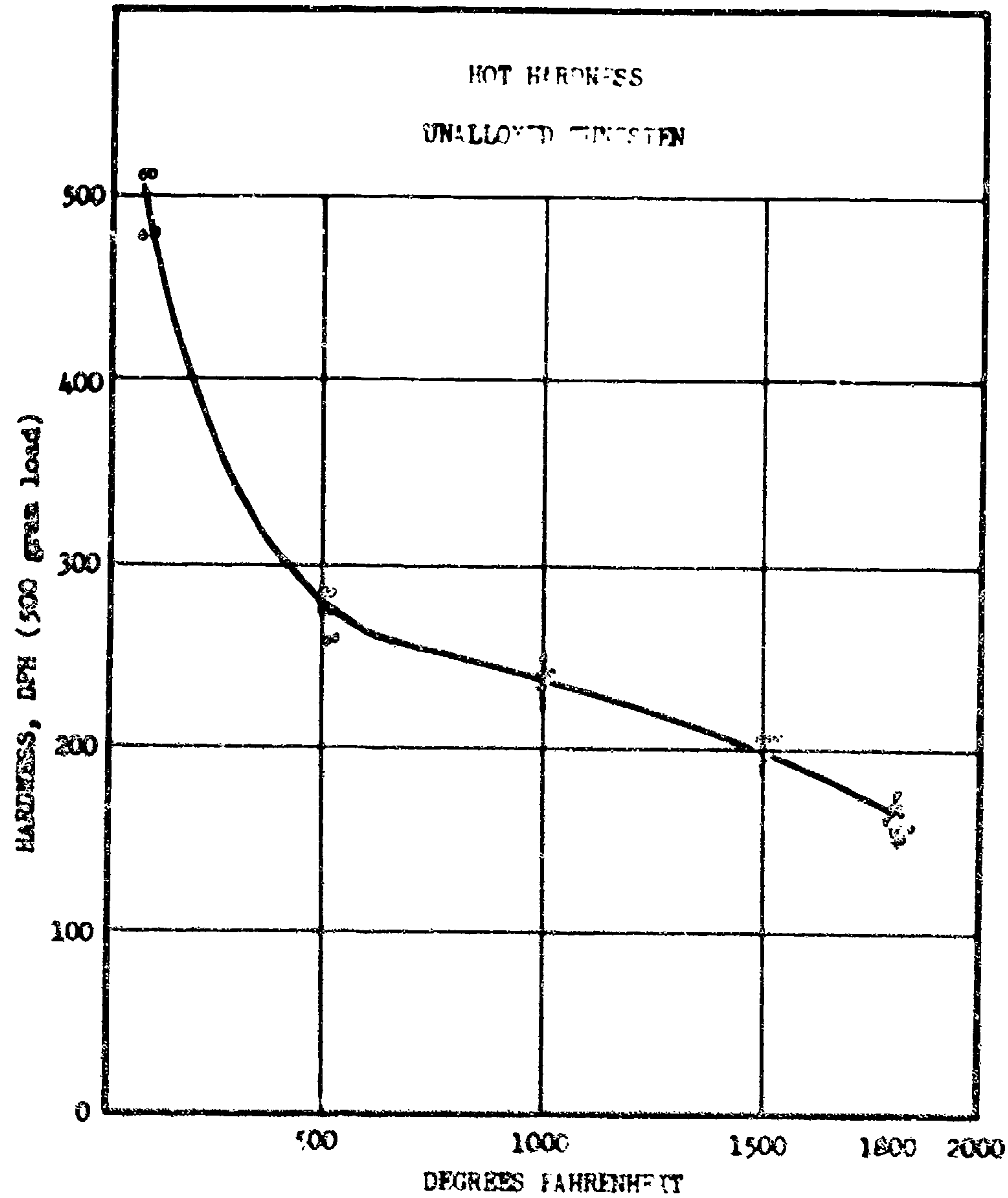

HOT HARDNESS
UNALLOYED TUNGSTEN
500
400
300
200
100
0
HARDNESS, DPH (500 gram load)
500
1000
1500
1800
2000
DEGREES FAHRENHEIT

FIGURE 14

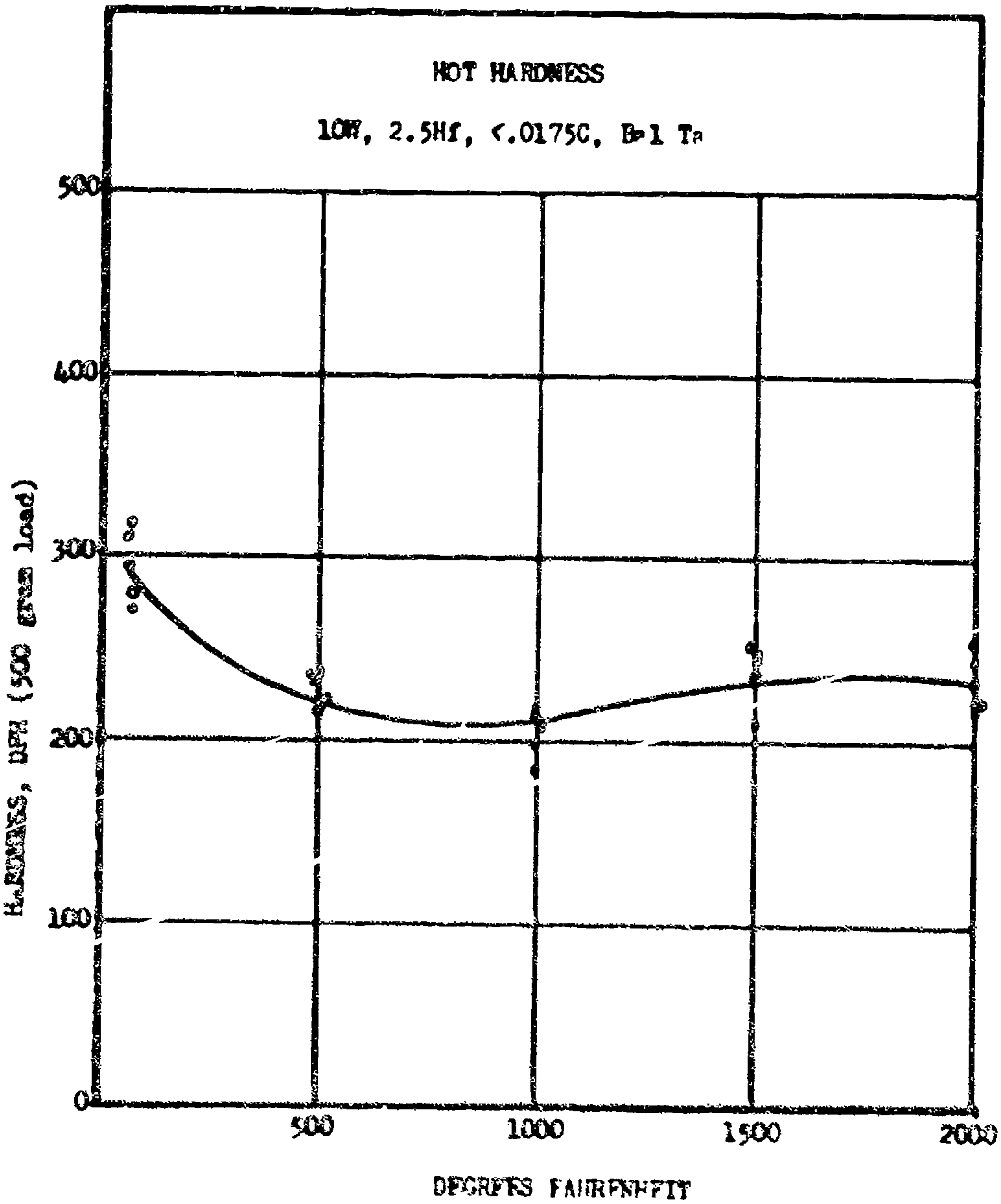
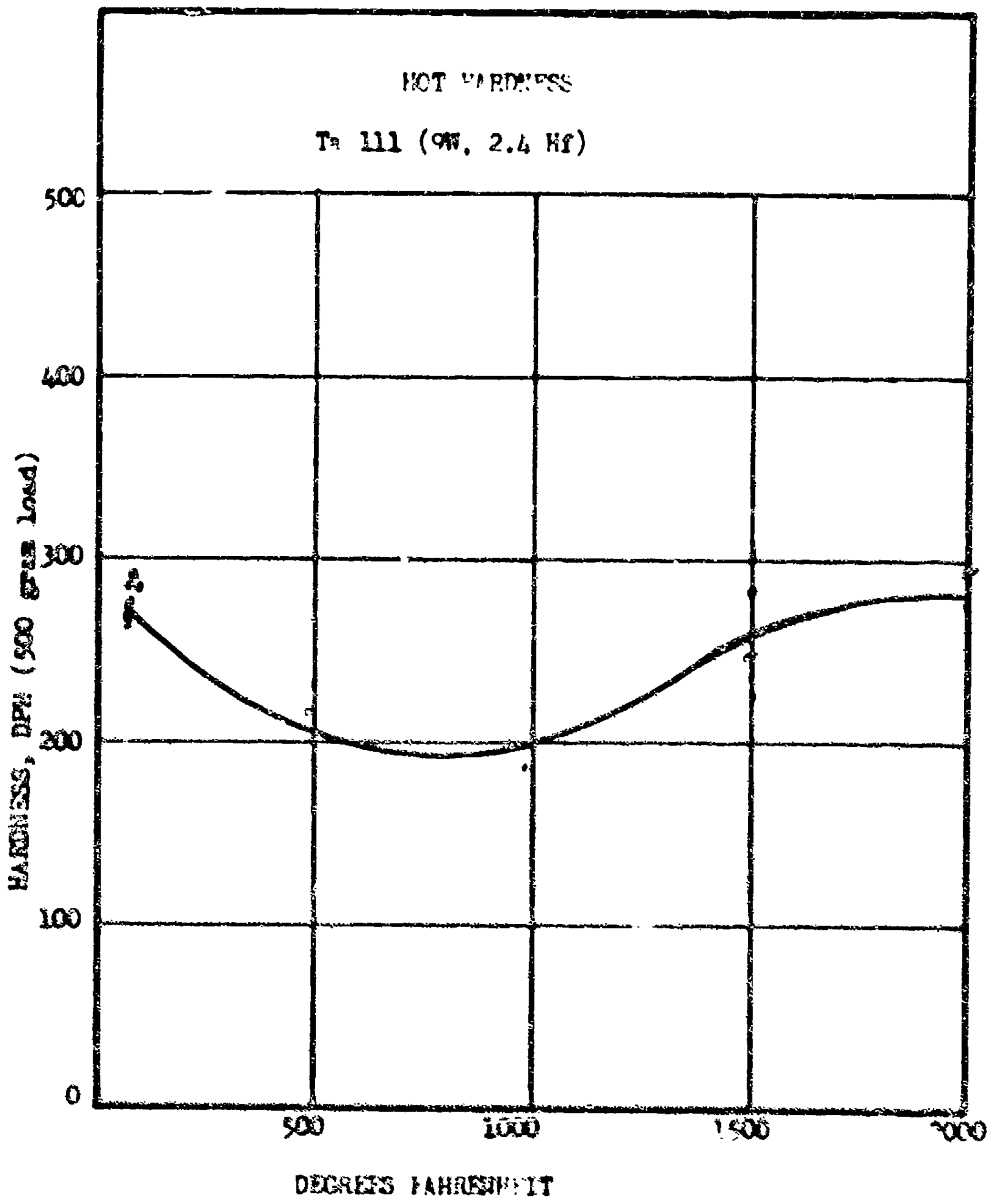

HOT HARDNESS
10W, 2.5Hf, <.0175C, B-1 Ta
500
400
300
200
100
0
500
1000
1500
2000
DEGREES FAHRENHEIT

FIGURE 15



HOT HARDNESS
Ta 111 (9W, 2.4 Hf)
500
400
300
200
100
0
HARDNESS, DPH (500 gram load)
500
1000
1500
2000
DEGREES FAHRENHEIT

FIGURE 16

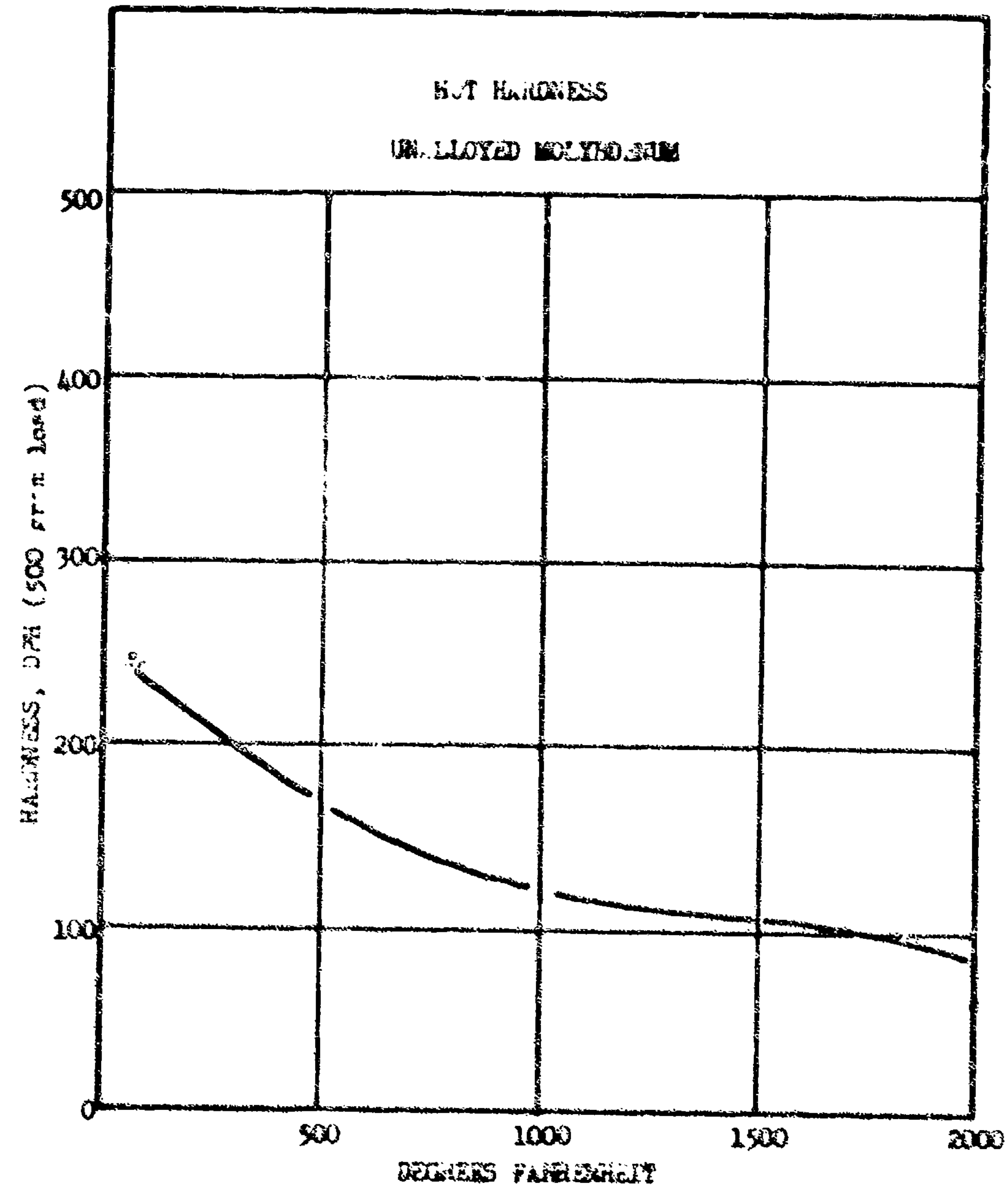

HOT HARDNESS
UNALLOYED MOLYBDENUM
HARDNESS, DPH (500 gram load)
0
100
200
300
400
500
500
1000
1500
2000
DEGREES FAHRENHEIT

FIGURE 17

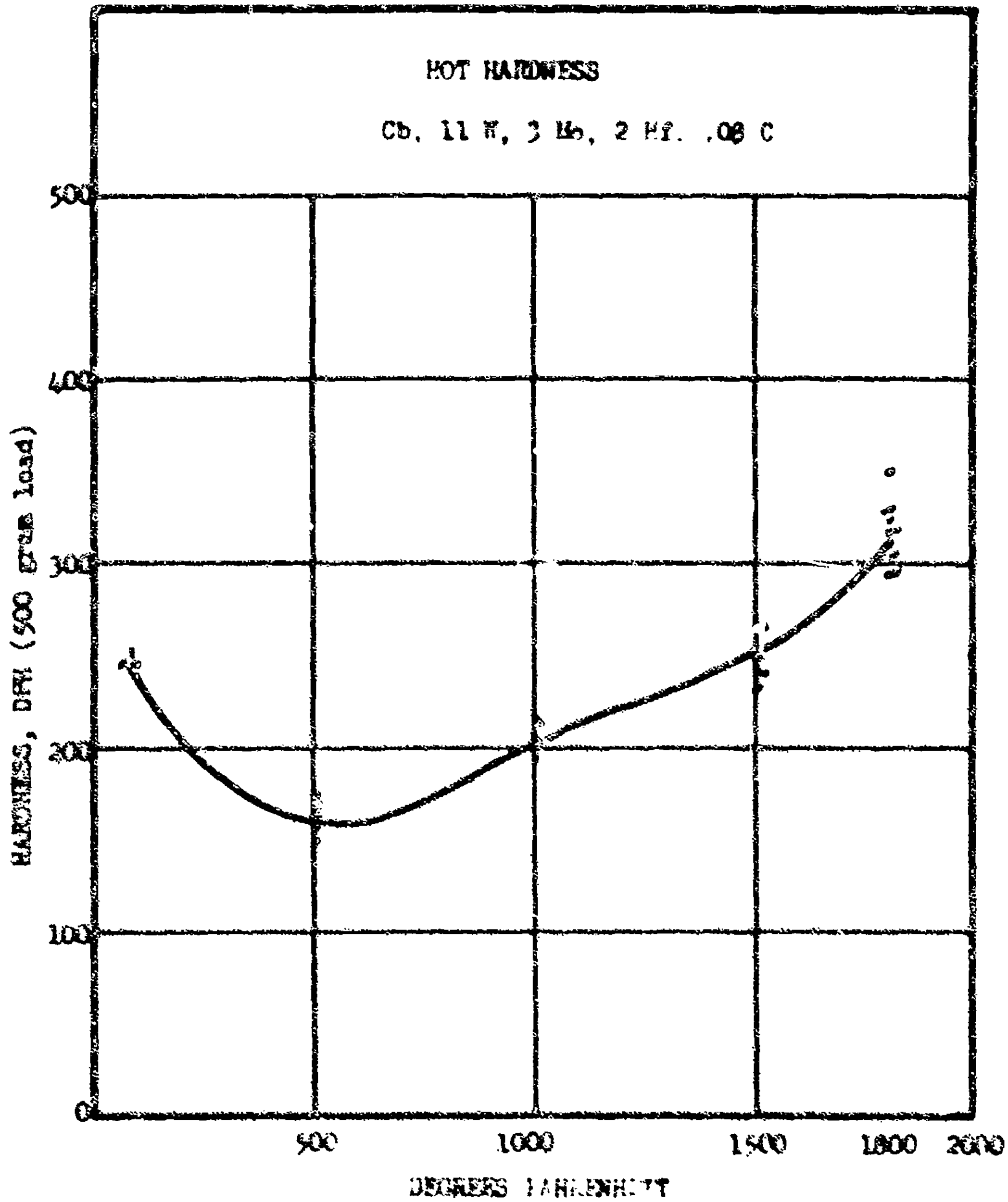

HOT HARDNESS
Cb, 11 W, 3 Mo, 2 Hf, .08 C
HARDNESS, DPH (500 gram load)
0
100
200
300
400
500
500
1000
1500
1800
2000
DEGREES FAHRENHEIT

FIGURE 18

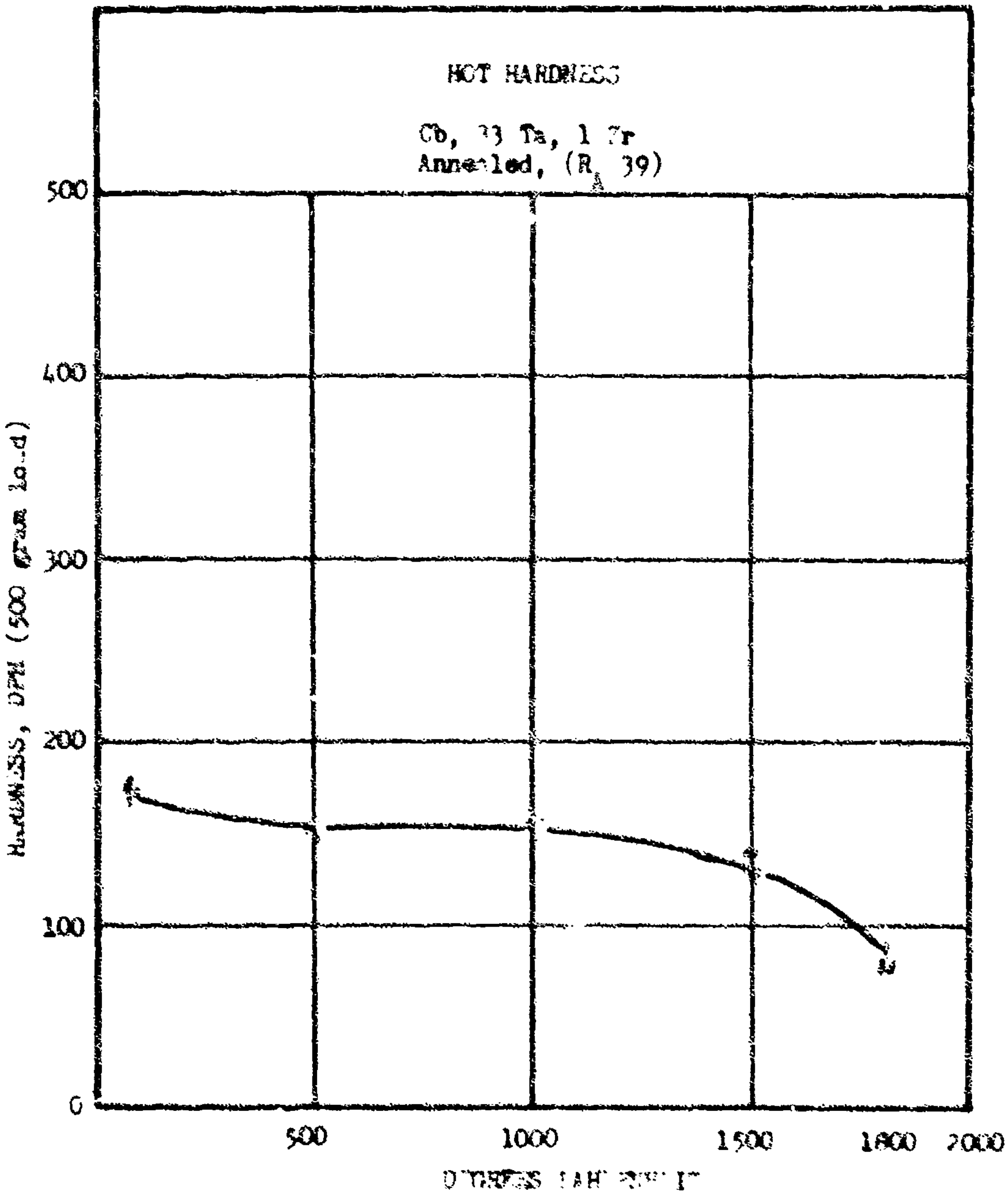


FIGURE 19

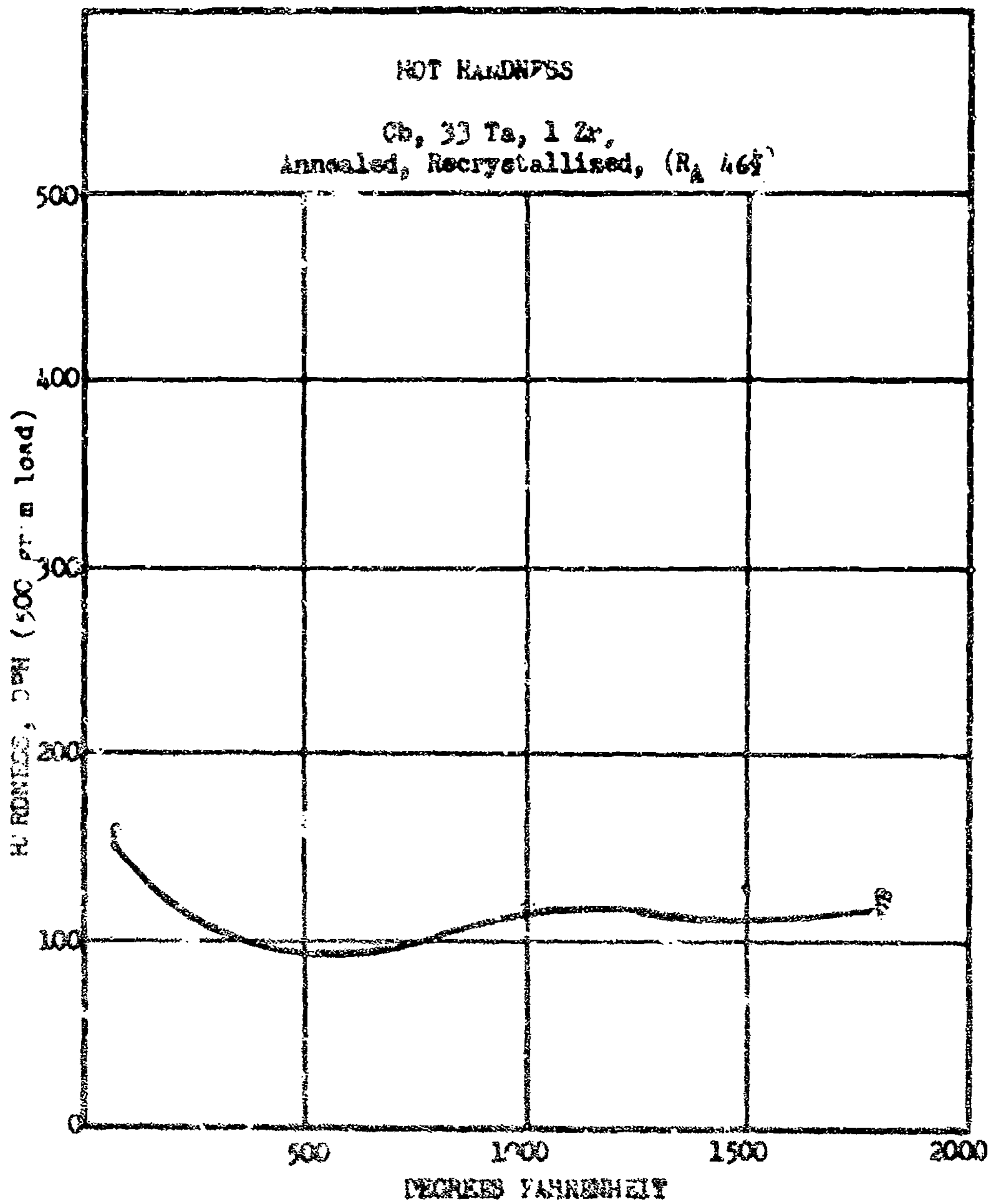
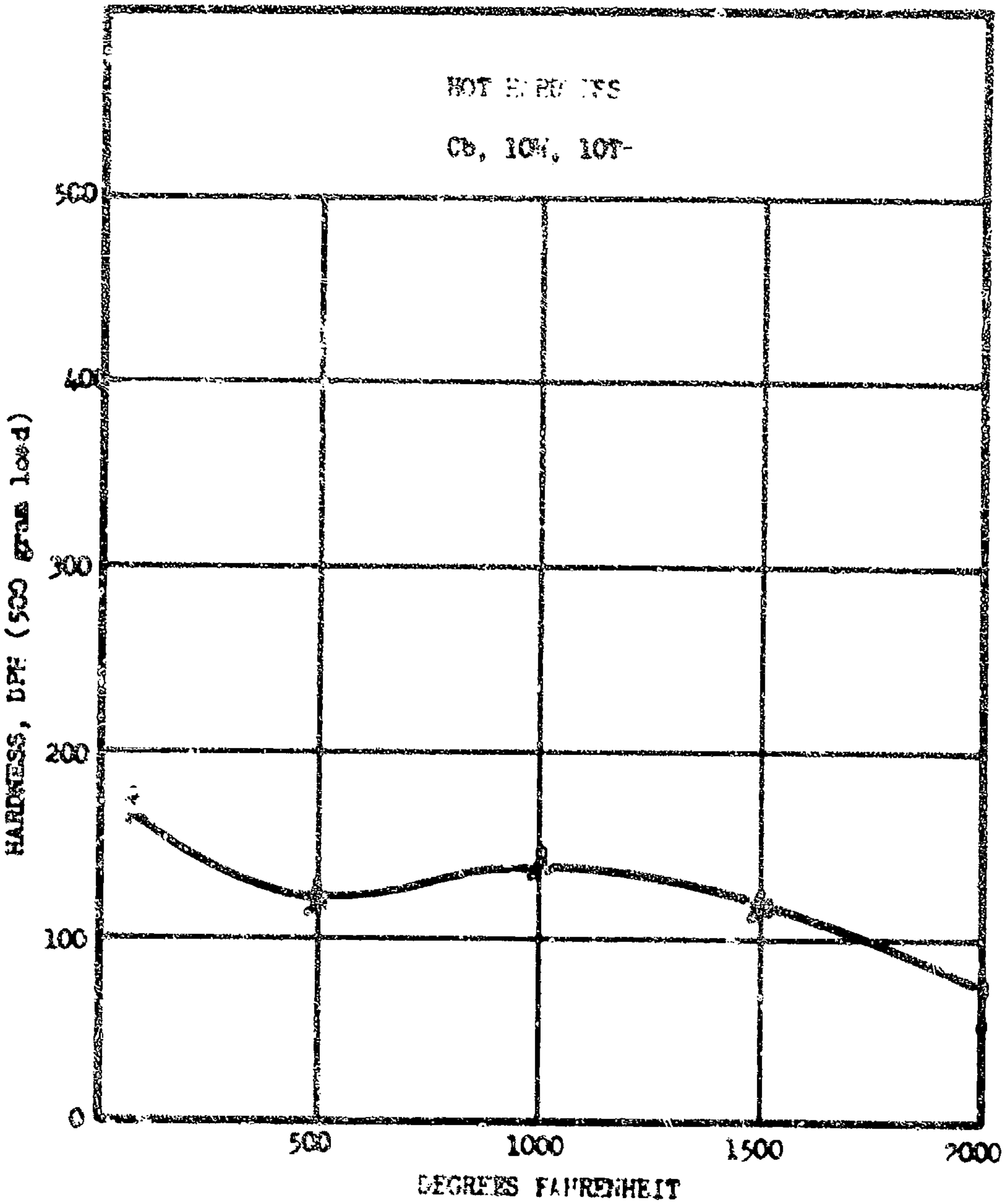

HOT HARDNESS
Cb, 33 Ta, 1 Zr,
Annealed, Recrystallized,
500
400
300
200
100
0
500
1500
2000
DEGREES FAHRENHEIT

FIGURE 20



HOT H RD SS
Cb, 10W, 10T-
500
400
300
200
100
0
HARDNESS, DPH (500 gram load)
500
1000
1500
2000
DEGREES FAHRENHEIT

FIGURE 21

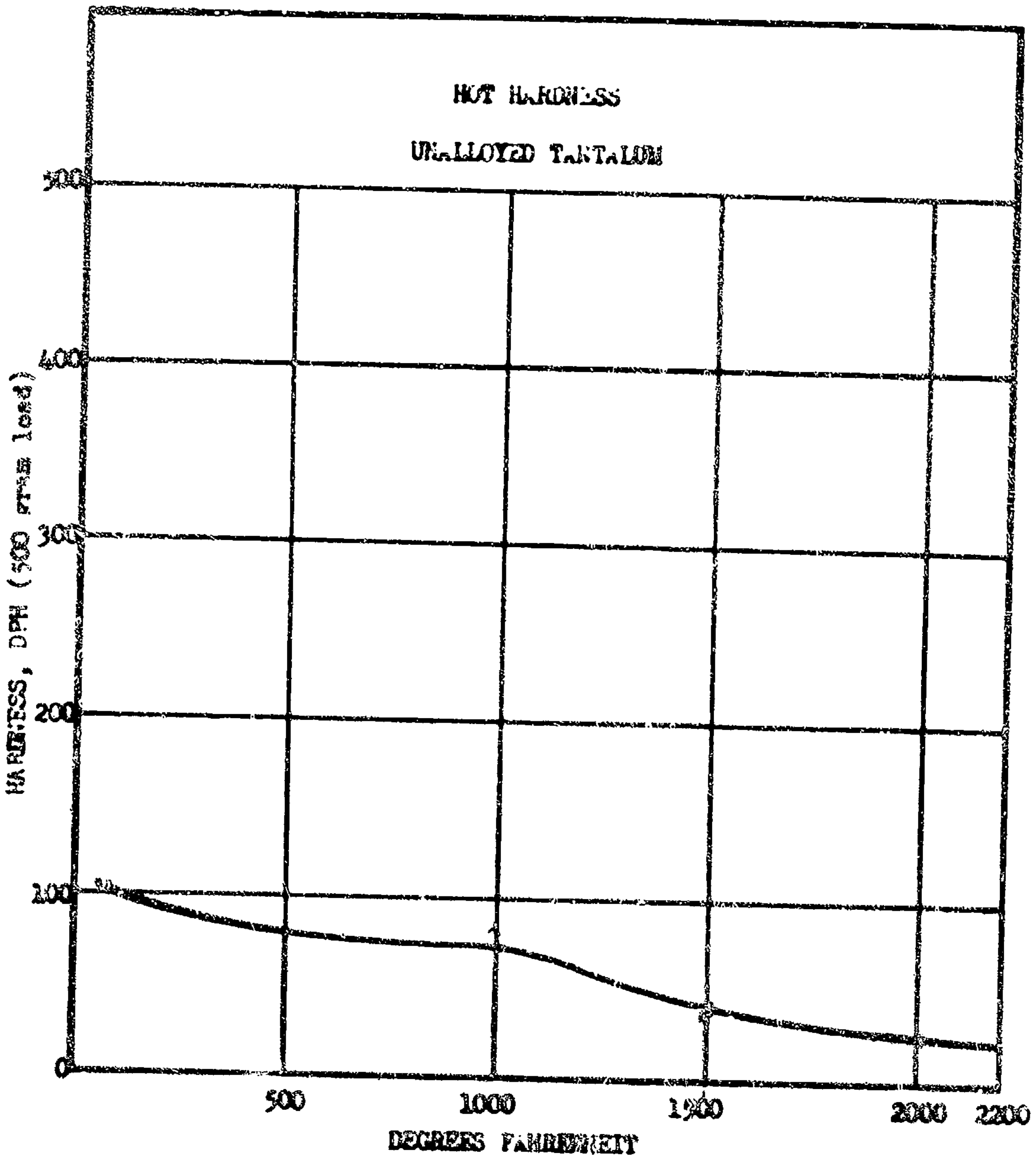

HOT HARDNESS
UNALLOYED TANTALUM
500
400
300
200
100
0
HARDNESS, DPH (500 gram load)
500
1000
1500
2000
2200
DEGREES FAHRENHEIT

FIGURE 22

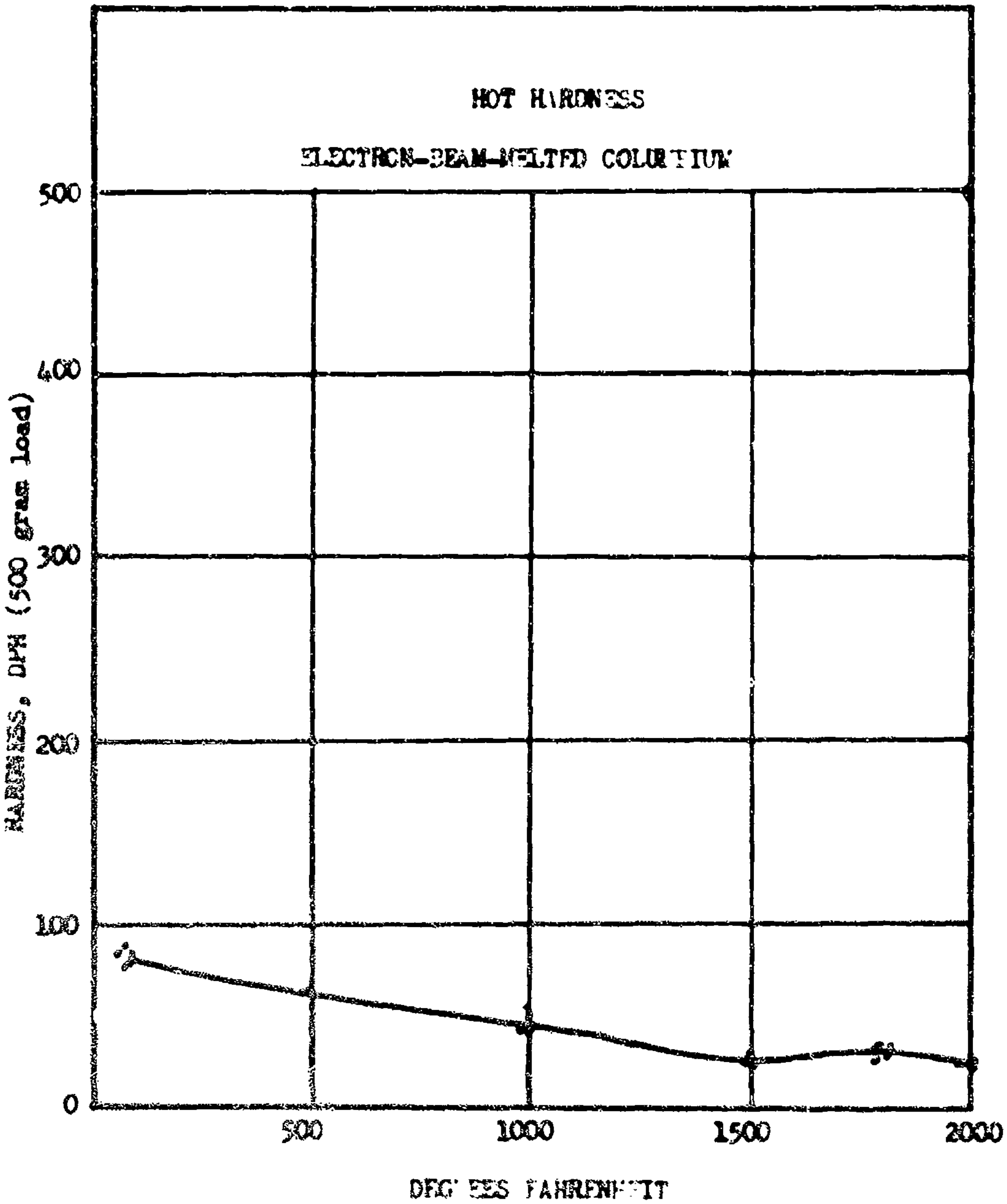

HOT HARDNESS
ELECTRON-BEAM-MELTED COLUMBIUM
HARDNESS, DPH (500 gram load)
500
400
300
200
100
0
500
1000
1500
2000
DEGREES FAHRENHEIT

FIGURE 23

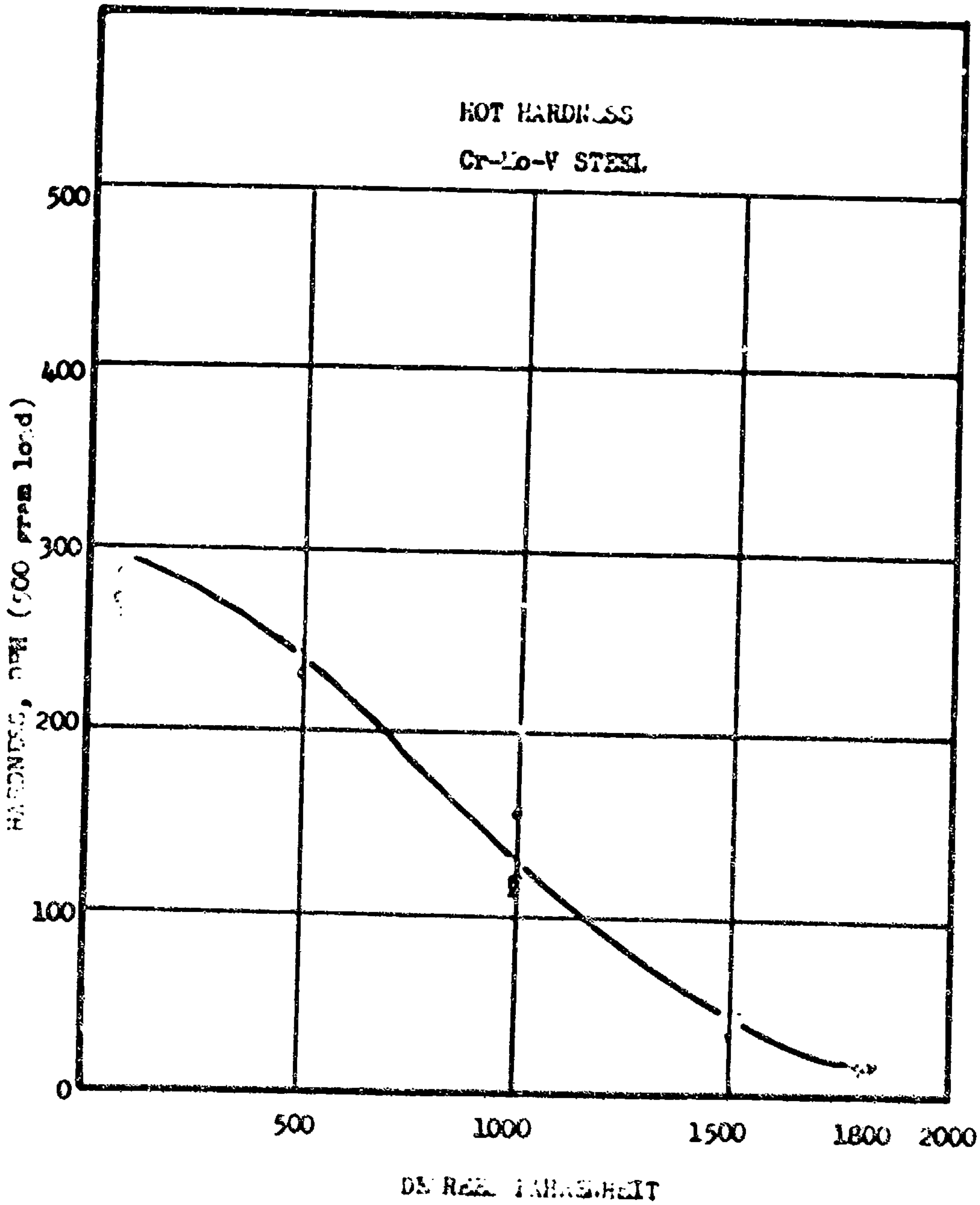

HOT HARDNESS
Cr-Mo-V STEEL
500
400
300
200
100
0
500
1000
1500
1800
2000

FIGURE 24

The hardness of tungsten (Figure 14) dropped quickly from approximately 500 DPH (R_C 49) at room temperature to 275 DPH (R_C 27) at 500°F, but much less rapidly to 163 DPH, (below R_C 20) at 1800°F. Tungsten was harder than any of the other materials tested except chromium up to about 1100°F and approximately equals the hardness of Su-16, Ta-111, and Ta-222 refractory alloys at about 1300°F.

These latter three alloys showed increases in hardness with increasing temperature after initially dropping in hardness (Figures 15, 16, 18). The unalloyed metals generally showed a continuing downward trend in hardness with increasing temperature although some regions existed in which hardness was relatively constant.

The chromium-molybdenum-vanadium steel used for gun barrels was harder than most of the refractory materials (except tungsten) at room temperature and at 500°F. Above 500°F, however, the hardness dropped rapidly with increasing temperature. Above 1000°F, it was softer than any of the other materials except tantalum and columbium. At 1800°F, it was softer than even these two metals.

CONCLUSIONS

Several of the refractory alloys tested especially the columbium-base alloy Su-16 and the two tantalum-base alloys Ta-111 and Ta-222 retained comparatively high levels of hardness to 1800°F or higher. Provided other requirements are met, these alloys appear promising for high-temperature applications such as gun barrel liners.

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